Experimental investigation of methanol crossover evolution during direct methanol fuel cell degradation tests

A. Casalegno*, F. Bresciani, M. Zago, R. Marchesi

Politecnico di Milano, Department of Energy, via Lambruschini 4, 20156 Milano, Italy

HIGHLIGHTS

- The methanol crossover during different DMFC degradation tests decreases.
- The water transport during different DMFC degradation tests remains nearly constant.
- The methanol crossover decrease in time presents both temporary and permanent contributions.
- The methanol crossover evolution could mitigate the degradation effect on DMFC efficiency.

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ABSTRACT

Methanol crossover and severe degradation are two of the most critical issues hindering the commercialization of direct methanol fuel cells. The experimental investigations found in the literature show that degradation has both permanent and temporary contributions; the latter can be recovered thanks to a suitable operation interruption. This work reports the experimental characterization of methanol crossover and water content in cathode exhaust during different degradation tests performed in continuous and cycling operation modes. Such investigation evidences a reduction of methanol crossover during the DMFC degradation tests that can be partially restored. Methanol crossover reduction presents both temporary and permanent contributions: the latter could be related to membrane degradation. Moreover the effect of both methanol crossover and electric power reduction on fuel cell efficiency are discussed.

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1. Introduction

Direct methanol fuel cell (DMFC) is a very promising technology as a power source for portable and uninterruptible power supply applications, due to the direct use of a high energy density liquid fuel [1–3]. However, the fuel is also the cause of its main drawbacks: lower efficiency and performance than polymer electrolyte membrane fuel cell (PEMFC) fed with hydrogen. This is due to the slower electrochemical methanol oxidation and to methanol crossover through the polymeric membrane.

In the literature methanol crossover has been extensively studied both experimentally and theoretically [4–8]. It has been widely demonstrated that it is closely related to membrane structure, morphology and thickness and to DMFC operating conditions such as temperature, pressure, and methanol concentration. Nowadays the main research activities are focussing on developing innovative materials to reduce methanol crossover [9,10], in order to reach market competitiveness. However other technical issues must be still overcome to enter into the market, among which the severe performance degradation.

In the literature the experimental investigations of DMFC degradation are continuously increasing [11–18], but generally, they focus only on catalyst characterization before and after the degradation test [12–15]. DMFC performance loss can be partially recovered interrupting the operation for diagnostics or by utilizing the appropriately developed procedures. Cha et al. characterize this behaviour and define a permanent and temporary contributions [17]. The former is due to irreversible degradation mechanisms, such as catalyst agglomeration, ruthenium dissolution and membrane delamination, while the latter is not fully understood nor systematically investigated. In Refs. [19–22] some operating strategies are presented in order to reduce the effect of temporary degradation, but the influence of operating conditions is seldom investigated.

Indeed methanol crossover is a technical issue of fundamental significance, because it determines a strong reduction of cathode

* Corresponding author.
 E-mail addresses: andrea.casalegno@polimi.it, casalegno.andrea@gmail.com (A. Casalegno).

potential, lowering DMFC performance, and a waste of fuel, decreasing system efficiency [23,24]. The analysis of methanol crossover behaviour during degradation tests is crucial, because the variation of both performance and efficiency is closely related to such phenomenon. In the literature there is a lack of *in situ* mass transport measurements during DMFC durability tests. Both methanol crossover and water transport in DMFC strongly depend on membrane and gas diffusion layer (GDL) properties [25,26] that may fade during operation. In Ref. [27] the analysis of water transport during a durability test is presented, but up to our knowledge no work proposes an investigation of methanol crossover evolution during degradation tests.

This work aims to investigate the evolution of methanol crossover during degradation tests, performed on two different Membrane Electrode Assemblies (MEAs), evaluating the influence of two operating strategies and anode methanol concentration. Moreover the effect of such methanol crossover variation on DMFC efficiency is investigated.

2. Experimental setup

2.1. Experimental equipment

The experimental setup for single cell DMFC characterization is described in Ref. [26], where the set of equations governing mass transport and the recent improvements to the experimental setup are explained in details. CO_2 measurement at cathode outlet is commonly accepted and used as a proxy for methanol crossover quantification. Data are acquired at a 0.5 Hz frequency and collected by mean of an *ad-hoc* developed Labview® software.

In this work electrochemical measurements are added to the experimental setup in order to improve the degradation characterization. An AC milliohmeter (TSURUGA 3566, estimated uncertainty: 1 mΩ cm^2) continuously measures the impedance of the fuel cell at 1 kHz single frequency, a reliable approximation of the membrane Ohmic resistance. The voltage of the fuel cell is simultaneously measured by the electronic load, the milliohmeter and also by a high precision acquisition board (NI 6218, estimated uncertainty: 1 mV) directly connected to the fuel cell, in order to have multiple readings.

Cathode exhaust composition has been measured by an SRA R-3000 μGC analyzer equipped by three columns and preceded by a liquid–gas gravity separator at room temperature. The first column is a nitrogen fed Molsieve for the hydrogen identification, the second one is a helium fed Molsieve for nitrogen, oxygen and CO identification while the third one is a helium fed PlotU for the CO_2 and light hydrocarbons identification such as methanol.

2.2. MEAs description

The first MEA used in this work is a commercial 22 cm^2 MEA: membrane is Nafion117, anode catalyst loading is 3 mg cm^{-2} (PtRu), cathode catalyst loading is 1.2–1.4 mg cm^{-2} (Pt). Anode diffusion layer is Sigracet® SGL10CA (thickness 400 μm, 10% PTFE content, without microporous layer), while cathode diffusion layer is Sigracet® SGL10CC (thickness 415 μm, 10% PTFE content, with microporous layer). During testing anode and cathode are fed respectively with 1.0 M methanol solution with stoichiometry equal to 3 and dry air with stoichiometry equal to 4. Nominal current density is 0.15 A cm^{-2}. The fuel cell temperature is kept at 60 °C by mean of a proportional, integral and derivative (PID) temperature controller.

Instead the second MEA used in this work is a commercial 25 cm^2 manufactured by IRD Fuel Cell A/S: membrane is Nafion115, anode catalyst loading is 1.8 mg cm^2 (PtRu), cathode catalyst

loading is 1.2–1.4 mg cm^2 (Pt). Both anode and cathode diffusion layer are Sigracet® SGL35DC (thickness 325 μm, 20% PTFE content, with microporous layer). During testing, unless differently indicated, anode and cathode are fed respectively with 1.0 M methanol solution with stoichiometry equal to 6 and air, saturated by water at ambient temperature, with stoichiometry equal to 3. Nominal current density is 0.25 A cm^{-2} and the fuel cell temperature is kept at 75 °C.

3. Continuous operation

3.1. Mass transport measurements on the first MEA

The test performed on the first MEA is composed of two continuous operation periods at constant current density interspersed with an interruption when diagnostic is performed, as reported in Fig. 1. The following procedure, named as "long refresh", is performed before each test: OCV is hold for few seconds with air feeding, then the air supply is switched off, while the methanol solution circulation is kept for 5 h. This procedure allows recovering most of the temporary degradation resulting in a considerable voltage recovery, but the reasons are still not fully understood [19,20,28].

In Fig. 1 the two components of the DMFC degradation can be clearly distinguished: the temporary one, that can be recovered, and the permanent one, that appears as a downward shift of the performances between the two tests.

Permanent degradation, calculated as in Ref. [22], is equal to 148 μV h^{-1}, a small part of the overall degradation, 770 μV h^{-1}, calculated as the difference between the initial and final voltages of the first test divided for the test duration; in fact the voltage drop associated with the first test is considerably higher than the downward shift between the two tests. Some researchers attribute DMFC temporary degradation to mass transport issues in cathode GDL and platinum oxide formation on cathode electrode [29]. An anode contribution to temporary degradation has been recently described in Ref. [30] and attributed to the reduction of methanol and water concentrations in the anode GDL and electrode.

Fig. 2 shows the measurements of CO_2 and water content in cathode exhaust during the two consecutive degradation tests; they highlight a decrease of CO_2 content during each test and a considerable recovery after operation interruption. Such reduction could be due to two possible reasons:
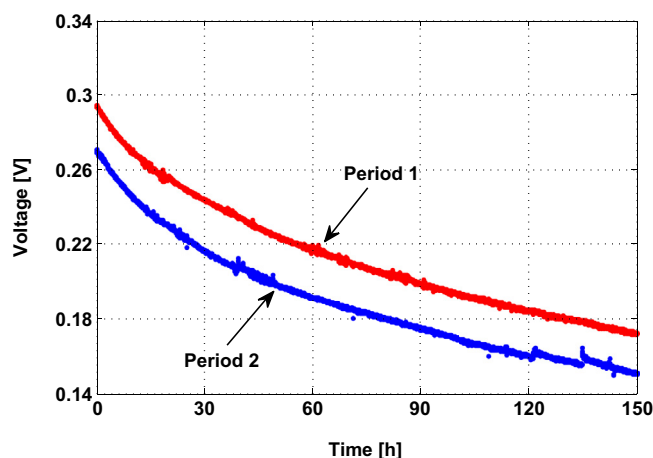

Fig. 1. Voltage decay during two consecutive tests in continuous operation performed on the first MEA at 0.15 A cm^{-2}.
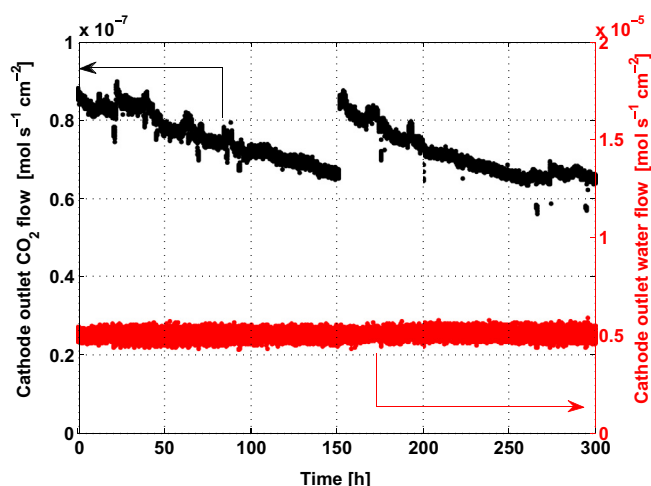
Fig. 2. CO_2 and water content at cathode outlet during the continuous degradation tests performed on the first MEA.

- a reduction of cathode effectiveness to methanol oxidation; in this case the effective methanol crossover could be constant, while in the cathode outlet methanol content increases and CO_2 content decreases;
- a reduction of methanol concentration at the anode electrode due to CO_2 bubbles accumulation that can determine mass transport limitations [30–32]; since the methanol crossover is related to methanol concentration gradient across the membrane, the lower is the concentration at anode electrode, the lower is methanol crossover.

A considerable variation of membrane properties (i.e. electro-osmotic drag and diffusivities),[1] able to determine the observed reduction of methanol crossover, is excluded, because it can occur in such intensity exclusively during long-term tests and cannot be recovered so quickly and easily. Likewise cathode GDL degradation can be excluded since water content in cathode exhaust, mainly dependent on cathode GDL properties [25], remains approximately constant during the tests. Analogously a considerable anode GDL degradation is improbable: it usually determines a reduction of material hydrophobicity [34] and an increase of diffusivity, that would determine an increase of methanol crossover.

As a consequence two possible explanations for the recovery of CO_2 content after the diagnostics are plausible; they are coherent respectively with the two previously proposed interpretations:

- the refresh procedure performed during the interruption determines a strong increase in cathode performance for two different reasons: the first period with air supply at OCV allows removing water flooding, that hinders cathode electrode performance; the second period without air supply permits to decrease the cathode potential under 0.5 V, allowing the reduction of platinum oxides with a consequent recover of platinum active surface [29];
- the interruption determines a progressive removal of CO_2 bubbles that hinder the correct operation of the anode electrode, determining an increase of methanol concentration at the anode and the consequent increase of methanol crossover [30].

A more detailed analysis has been carried out on the second MEA, previously described, to elucidate further these phenomena.

3.2. Mass transport measurements on the second MEA

Thanks to experimental setup improvements, the second MEA has been characterized more completely, including the analysis of cathode exhaust composition.

Two consecutive continuous operation tests have been performed at two different methanol concentrations, keeping the mixture flow constant (1 M during period 1 and 2 M during period 2), in order to investigate the effect of anode methanol concentration on crossover evolution, as reported in Fig. 3.

During the first period the cell has been operated for 50 h with 1 M concentration. As previously CO_2 and water content in cathode outlet are continuously measured; furthermore cathode exhaust is connected to the gas chromatograph where the dry composition is continuously analysed. From the exhaust composition acquired during period 1, Fig. 4, it is possible to highlight the following key points:

- the CO_2 content in cathode outlet decreases during operation, while water content remains approximately constant, coherently with the previous test;
- the amount of unconsumed methanol detected in the cathode exhaust is very limited and remains almost constant during the test[2];
- the oxygen content in cathode outlet progressively increases.

These results permit to state that methanol crossover effectively decreases during the DMFC continuous operation, implying a reduction of CO_2 content in cathode exhaust, a constant methanol content and a reduction of oxygen consumption. Therefore CO_2 content in cathode outlet is confirmed to be a proper proxy for methanol crossover also during degradation tests. Moreover, a substantial recoverability of both voltage degradation and methanol crossover decrease has been verified, confirming the results obtained with the first MEA.

Consequently the most probable origin of such methanol crossover reduction is the second possibility previously mentioned: the decrease of local methanol concentration in the electrode, caused by gas-phase CO_2 accumulation within electrode and GDL, as recently proposed in Ref. [30].

Doubling the methanol concentration, period 2, DMFC performance is hindered. Since methanol crossover is proportional to methanol concentration in the anode electrode and considering that the air stoichiometry is kept constant during all the tests, it is clear that oxygen transport limitations can occur at cathode due to a more intense methanol oxidation.

The exhaust composition results related to period 2, Fig. 5, are coherent with the proposed interpretation:

- the CO_2 content in cathode exhaust is significantly higher than in the previous test coherently with the doubled anode methanol concentration; it significantly decreases in absolute value, but the relative decrease is lower than in the previous case;
- the oxygen content is considerably lower than in the previous case coherently with a strong increase in methanol crossover; it increases during the test as the CO_2 content decreases;

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[1] Affected also by the capability to stay effectively hydrated, therefore related to membrane crystallinity and extension of the hydrophilic ionic zone [33].

[2] It is necessary to underline that methanol concentration in the cathode exhaust is significantly lower than the saturation value at ambient temperature, confirming the measurement reliability.
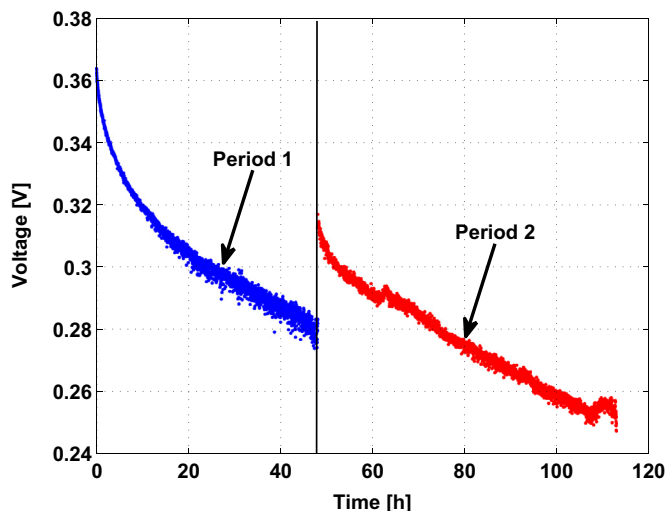
Fig. 3. Voltage decay during two consecutive tests in continuous operation, performed on the second MEA at 0.25 A cm^{-2}: period 1 at 1 M, period 2 at 2 M.

- the unconsumed methanol content is higher than in the previous case but still not relevant; it remains almost constant during the operation.

3.3. Effect on DMFC efficiency

Despite the continuous operation is not the common way to operate a real DMFC system, it is interesting to compare the influence of performance and methanol crossover reductions on the DMFC efficiency as defined in Ref. [23], considering unconsumed fuel recirculation:

$$\eta = \frac{V \cdot i}{\left(N_{CH_3OH}^{a,cons} + N_{CH_3OH}^{crossover}\right) \cdot LHV_{CH_3OH}} \qquad (1)$$

where the converted fuel flow includes the fractions used in the anode electrochemical reaction $N_{CH_3OH}^{a,cons}$ and wasted with methanol crossover at the cathode $N_{CH_3OH}^{crossover}$.

In this work the fuel recirculation is not really performed in order to avoid fuel contaminant accumulation that could have an unpredictable effect on degradation tests.

Table 1 shows a comparison among the variations of power, methanol crossover and efficiency during tests. Period 1 is characterized by a crossover reduction that is more intense than the power one; this determines a less pronounced reduction of DMFC efficiency compared to power one.

During period 2 power and crossover reductions are less severe than during period 1; in this case the positive effect of crossover reduction on efficiency appears less evident.

This analysis permits to conclude that in some conditions methanol crossover evolution, determining a reduction of cathode parasitic loss and methanol wasting, can partially mitigate the effect of performance degradation on DMFC efficiency, in fact the voltage decrease, caused by membrane and electrodes degradation, still remains the dominant effect.

4. Cycling operation

4.1. Description of the operating strategy

The literature presents different operating strategies to minimize the temporary degradation that, adopting continuous operation, leads to a dramatic reduction of DMFC power, as previously discussed. For this reason, DMFCs are often coupled with batteries to cover load peaks and operation interruption, ensuring power continuity for the user.

Among different possible operating strategies, one suggested by IRD Fuel Cell A/S has been chosen: it consists in 20 min of operation interspersed by 1 min refresh procedure. The latter is composed of a sequence of OCV and cathode air feeding interruption,[3] similar to that reported in Ref. [20], that allows recovering the temporary degradation.

During the OCV period, after the air feeding is switched off, the cathode potential drops to less than 0.3 V and, when the operation restarts, a significant positive effect on performances is obtained.

The reason of such refresh effect on temporary degradation is still not fully understood. The following interpretation can be proposed. The OCV period reduces the cathode flooding, because of the temporary absence of electro-osmosis and water production, as also reported in Ref. [19]. Furthermore, when the air feeding is interrupted, oxygen is depleted by methanol crossover leading to a strong decrease of cathode potential, resulting in a reduction of cathode platinum oxides, that were previously formed during operation at high potential [29]. Moreover during operation interruption, the gas-phase CO_2 accumulated in anode GDL and electrode is removed [30].

Fig. 6 shows the degradation test performed on the second MEA; it is composed of two test periods in similar conditions: period 1 and period 2, interspersed by a long refresh, as previously described. Such procedure still permits a considerable performance recovery: the voltage value at the beginning of period 2 is considerably higher than that at the end of period 1, suggesting that the adopted operating strategy is not sufficient to fully recover temporary degradation. In both cases the appearing degradation rate is about 320 μV h^{-1}, calculated with the method reported in Ref. [22].

Polarization curve tests, including mass transport measurements, have been performed before and after the whole degradation test, in order to evaluate the permanent degradation,[4] Fig. 7. A difference of 40 mV between the two curves, at the nominal current density of 0.25 A cm^{-2}, is calculated; the resulting permanent degradation rate is equal to 123 μV h^{-1}, much lower than the appearing degradation.

4.2. Mass transport analysis

The measurements of methanol crossover, during the above mentioned tests, are reported in Fig. 8: methanol crossover tends to decrease during period 1, then, during the interruption, it recovers and decreases again during period 2.[5] This result is coherent with the continuous operation ones, but the decrease of methanol crossover now is less pronounced, confirming a correlation between temporary degradation and methanol crossover decrease. Fig. 9 shows the methanol crossover values measured during the two polarization curves reported in Fig. 6: a reduction of methanol crossover in reference conditions is evident.

As a consequence the reduction of methanol crossover during the DMFC operation appears determined by two contributions, similarly to voltage degradation: a temporary one, that can be recovered by refilling the anode with fresh mixture during the operation interruption, and a permanent one, that could be

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[3] The details cannot be provided for confidential agreement.

[4] The polarization curve test is performed after a long refresh procedure in order to nullify the effect of accumulated temporary degradation.

[5] Unfortunately a testing setup failure determined a period of improper operating conditions about the 240th hour, hindering the reliability of period 2 data.
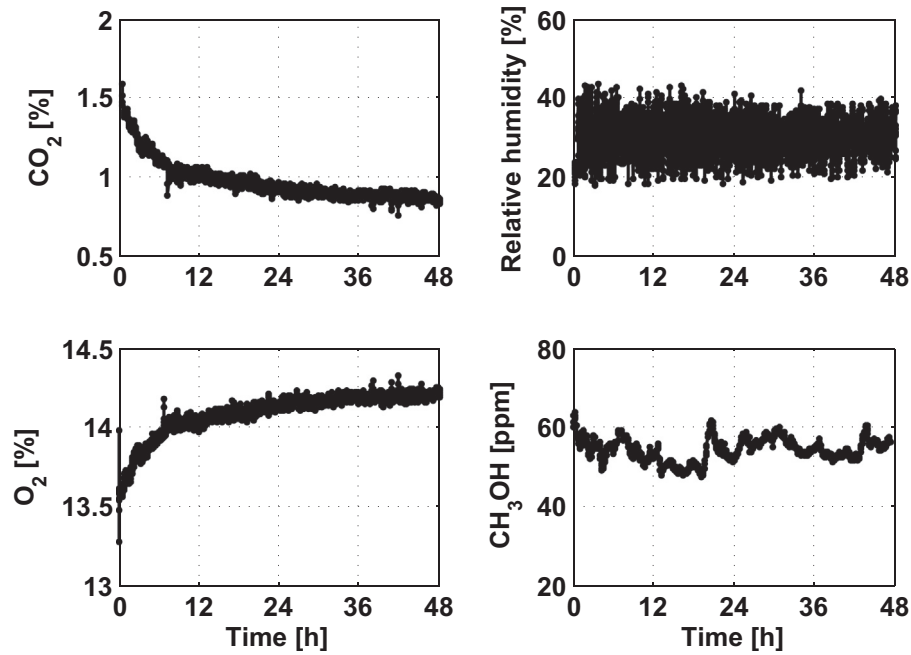
Fig. 4. Cathode outlet composition analysis during continuous operation test during period 1 (1 M).

indicative of a membrane alteration. The latter, in accordance with the measurements reported in Refs. [11,12], can be related to a loss of ionomeric groups and an increase in crystallinity, that implies a reduction of hydrophilic proton conductive zone, and can be already evident after 200 h of operation.

Methanol crossover evolution is related to two main factors: methanol concentration at the anode electrode and transport properties of the electrolytic membrane. Since polarization curves are comparable and acquired in reference conditions, a similar methanol concentration profiles at the electrode can be reasonably assumed; therefore the permanent reduction of methanol crossover is probably related to a modification of

membrane properties. This is confirmed by an increase of membrane ionic resistance, measured by the AC milliohmeter: respectively equal to 155 mΩ cm^2 and 191 mΩ cm^2 at beginning and end of test. Note that no significant membrane properties modification is observed in the previous tests, probably because the first MEA presented a thicker and more stable membrane, while the continuous tests on the second MEA have a too short duration.

Hence, by applying the Ohm's law, it is possible to calculate the potential drop that can be associated to the membrane degradation: 9 mV, less than 25% of permanent degradation calculated from polarization curves.
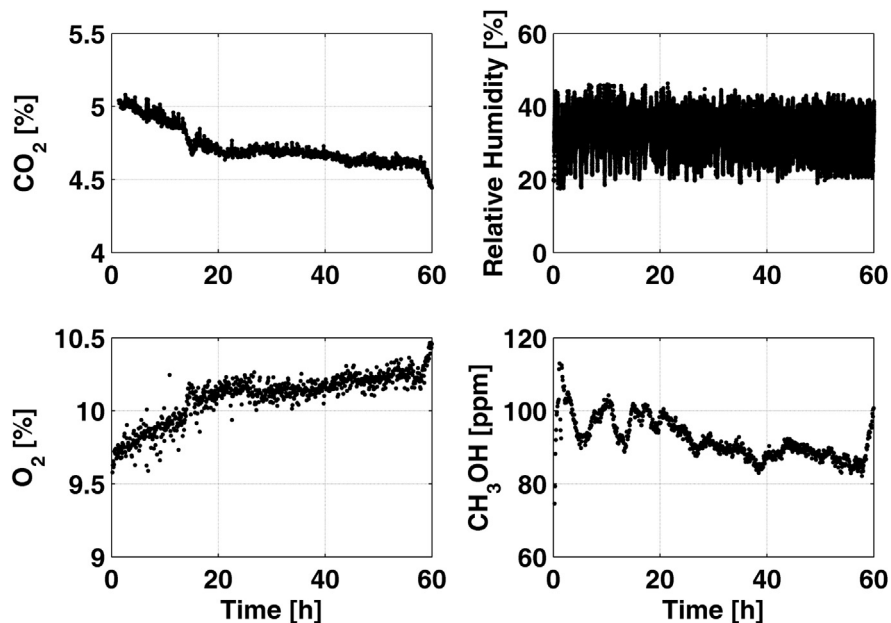

Fig. 5. Cathode outlet composition analysis during continuous operation test during period 3 (2 M).

Table 1
Summary of power, methanol crossover and efficiency reduction during continuous operation at 1 M and 2 M.

	After 2 h	After 20 h	After 50 h
1 M (period 1)			
Power reduction [%]	100	87	80
Crossover reduction [%]	100	72	61
Efficiency reduction [%]	100	92	86
2 M (period 2)			
Power reduction [%]	100	93	85
Crossover reduction [%]	100	93	90
Efficiency reduction [%]	100	94	86

Fig. 6. Voltage decay during two consecutive tests in cycling operation performed on the second MEA at 0.25 A cm^{-2}.

Once the contribution of membrane degradation is quantified and the degradation of the GDLs is assumed negligible, more than 75% of permanent degradation can be attributed to the electrodes.

The DMFC efficiency reduction is comparable with power decrease, because the reduction of methanol crossover is appreciable but its effect is not very significant, similarly to the 2 M case of the first MEA.

Further ex-situ investigations are needed to identify the mechanisms that lead to membrane modification during operation
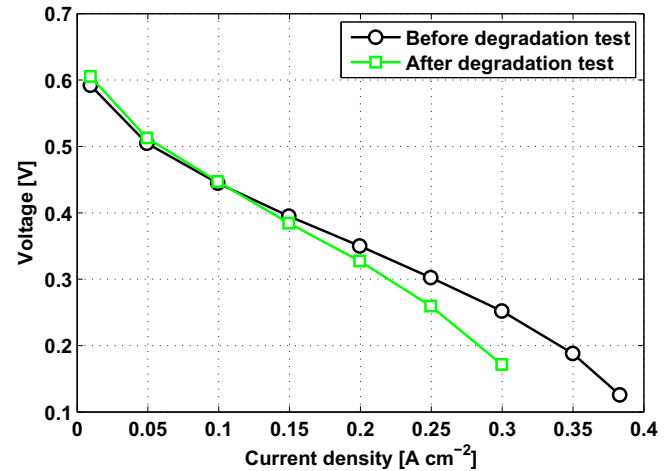

Fig. 7. Polarization curves at start/end of the degradation test (3.86 g min^{-1} of mixture at 1 M, 0.336 g min^{-1} of air).
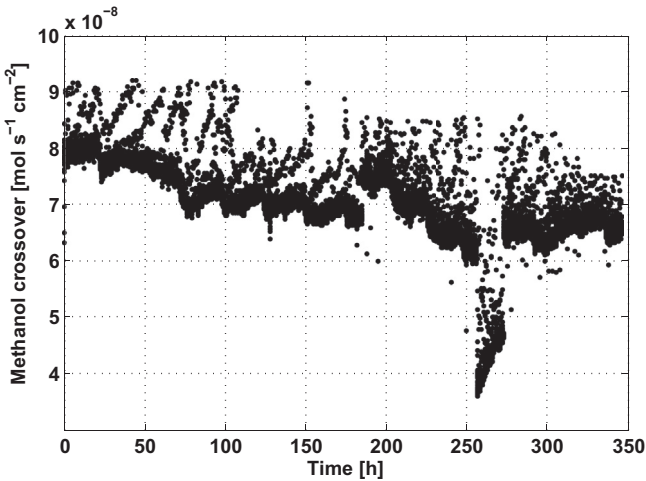

Fig. 8. Methanol crossover evolution during the degradation test.
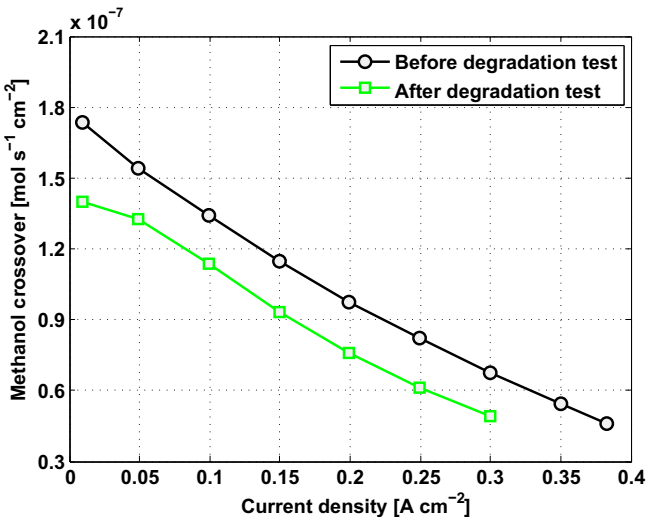

Fig. 9. Methanol crossover measured during polarization curves.

and to clarify the degradation mechanisms that occur at the anode and cathode electrodes.

5. Conclusions

This work presents an experimental investigation of methanol crossover during degradation tests, performed adopting different operating strategies on two different MEAs. The main conclusions that can be drawn are:

- the methanol crossover decreases considerably in continuous operation, while the reduction is less pronounced adopting a suitable operating strategy, coherently with the performance degradation;
- the water content at cathode outlet remains almost constant during degradation tests; therefore cathode GDL degradation seems to be negligible;
- the methanol crossover reduction could be associated to two different phenomena: a temporary one, probably due to a decrease of methanol concentration and CO_2 accumulation at anode electrode, and a permanent one related to membrane alteration;

- the methanol crossover reduction during degradation tests has a significant positive impact on DMFC efficiency, especially during operation with 1 M methanol feeding.

Further investigations are needed in order to clarify the relation between temporary degradation and methanol crossover reduction.

Acknowledgement

List of symbols

V	voltage (V)
i	current density (A cm^{-2})
N	specific molar flow (mol s^{-1} cm^{-2})
LHV	lower heating value (J mol^{-1})
η	efficiency

Subscripts
CH$_3$OH   relative to methanol

Superscripts
a, cons   relative to anode consumption
crossover relative to crossover

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